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IN THE UNITED STATES PATENT AND TRADEMARK OFFICERECEIVED  
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APPLICANT: Jawad Haidar ) Group Art Unit 7414  
 SERIAL NO: 10/560,804 ) Examiner: Zhu, Weiping  
 FILED: December 15, 2005 ) Docket: CU-4560  
 TITLE: A METHOD AND APPARATUS FOR THE PRODUCTION OF METAL  
 COMPOUNDS

MAY 01 2009

THE COMMISSIONER FOR PATENTS  
 P.O. Box 1450  
 Alexandria, VA 22313-1450

AFFIDAVIT UNDER 37 CFR 1.132

1. Jawad Haidar, the inventor named in the application for letters patent of the United States for an invention entitled A METHOD AND APPARATUS FOR THE PRODUCTION OF METAL COMPOUNDS, Serial No. 10/560,804, filed December 15, 2005, declare as follows:

1) I am a scientist working for the Commonwealth Scientific and Industrial Research Organisation (CSIRO). CSIRO is the Australian Government's main national research laboratory and is classified by the International Scientific Institute (ISI) among the top research institutions in the world. Before joining CSIRO in 1990, I obtained a Doctorate degree and a Master degree from the University of Paris (France). My research career includes extensive work on electric arcs, electric discharges, modelling of arc-welding, plasma vapour deposition and materials synthesis. My work on arc discharges led to the discovery of important basic phenomena in arc physics and in the late 1990s I developed the first ever numerical simulation of droplet formation of arc welding. My experimental activities also included developing technologies for nanopowder production and physical vapor deposition of tetrahedral carbon. I regularly attend scientific conferences and receive and read numerous scientific journals. I am the sole inventor of the invention the subject of the present application.

2) I have been asked to review the patent specification for US patent no. 5,032,176 (Kometani et al) and describe its relevance (if any) to the invention the subject of the present application. The method disclosed by Kometani et al. does not use aluminium as a reducing agent. In col 12, lines 5-10, it is stated that the purpose of including aluminium with the reducing agent is to produce a titanium-aluminium composite powder, suggesting that the aluminium is not the reducing agent as it is

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conserved through the reduction process. Indeed, it is well known to me that reactions between titanium chlorides and magnesium (or sodium) are more favourable than reactions between titanium chlorides and aluminium. The Gibbs free energy for reacting  $TiCl_4$  with Al is -102 kJ/mole while this energy is -468 kJ for Mg and -800 kJ/mole for Na. This means that in the presence of Mg or Na,  $TiCl_4$  is unlikely to react with anything else.

3) Figures 1 and 2 below depict the results of calculations I have recently performed for the reactions which are theoretically possible for chemical compositions having a mixture of  $TiCl_4$  + 2 Mg + 2 Al in Figure 1 and  $TiCl_4$  + 4 Na + 2 Al in Figure 2. For Mg, Na and Al, the stoichiometric amount required to reduce 1 mole of  $TiCl_4$  are respectively 2 moles, 4 moles and 1.33 moles. Including excess Al in the calculations in Figures 1 and 2 is intended to show that the presence of excess Al does not improve reactivity between  $TiCl_4$  and Al. The results in Figures 1 and 2 were obtained using Outokumpu HSC chemistry, a renowned commercial package, and clearly show zero reactions between  $TiCl_4$  and Al. This confirms to me that, in the presence of Mg or Na,  $TiCl_4$  will not react with Al. The amount of  $AlCl_3$  formed in both cases is nil. Thus, it is my understanding that because Kametani et al require the presence of either Na or Mg, if Al were added for alloying with Ti to form a Ti-Al alloy, the Al would not react at all with  $TiCl_4$ .

4) To emphasise this point, in Figure 3 I show the results of a similar calculation for a composition including 10 moles of Al. Here, even with the presence of highly excessive amounts of Al relative to Na, there are absolutely no reactions between  $TiCl_4$  and Al. The results in Figures 1, 2 and 3 prove to me that for the method disclosed by Kametani wherein titanium tetrachloride is reduced by molten agent including Mg or Na, Al cannot be a reducing agent when present in the molten reducing agent.

5) I have also been asked to consider and describe what reactions would be likely to occur if Al were to act as a reducing agent in the methods described in Kametani. Kametani requires his reactions to occur at 650 C (The temperature required for  $TiCl_4$  throughout the specifications). Reactions between Al and  $TiCl_4$  at 650 C, is a one step process leading directly to formation of  $Al_3Ti$ (solid) and  $AlCl_3$ (solid) (as is depicted in Figure 3 of the article by Murphy which was also cited by the Examiner). As such, at a temperature of 650 C (~900 K), the reaction between  $TiCl_4$  and Aluminium would be a single step reduction process and not a two-step process as is the case in the present application. Our technology attempts to solve

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problems associated with the one step process, namely the formation of multi-phase compounds (Al<sub>3</sub>Ti) and AlTi as in Murphy (Figure 3).

6) I have also been asked to review a paper by Dr A. B. Murphy entitled 'Equilibrium calculations of the reduction of titanium tetrachloride by aluminium and hydrogen', published in *High Temperature Chemical Processes*, volume 3, pp. 365-374 (1994), and describe its relevance (if any) to the methods described in Kametani et al and to the invention the subject of the present application.

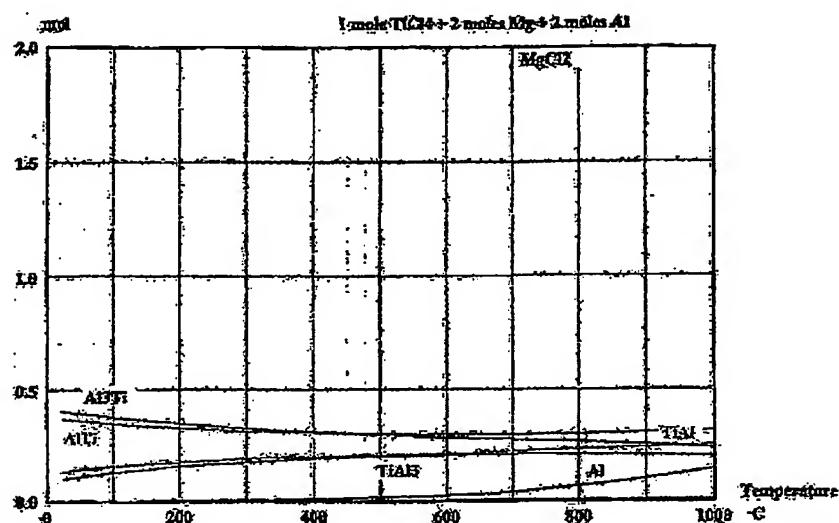
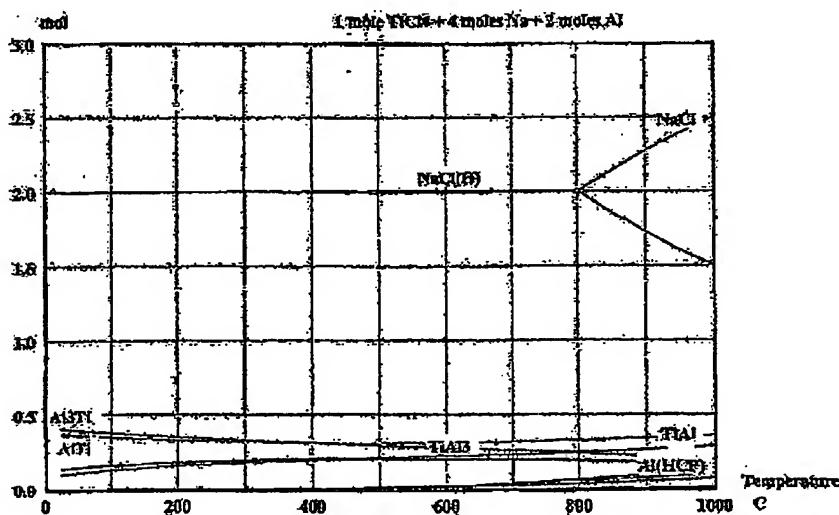
7) The results of Murphy are relevant only in extreme conditions at high temperatures in excess of 3000 K wherein all reactants are gaseous, as clearly explained in Section 4 of his paper. For the invention the subject of the present application, reactions typically start at less than 200 C (~450 K) and then the temperature is increased to 1000 C (1273 K). Also, for the present application in Stage 1 of the process, Al powder or flakes with a limited surface area are used, and Stage 2 of the process is purely based on solid-solid reactions. Thus, whilst the results of Murphy indicate that reactions between TiCl<sub>4</sub> and Al can occur, they are not directly relevant to the invention the subject of the present application.

8) I would not relate the results of Murphy as depicted in the paper mentioned above and the disclosure of Kametani for several reasons: a)- in the presence of Mg or Na as in Kametani, TiCl<sub>4</sub> would not react with Al, b)- the results of Murphy are not valid at the temperatures required by Kametani, and c)- Murphy clearly states that his results may be useful for applications in production of titanium-aluminium alloys only if the reactants are heated to high temperatures and then cooled to condense the resulting gases; this goes opposite to the teaching of Kametani.

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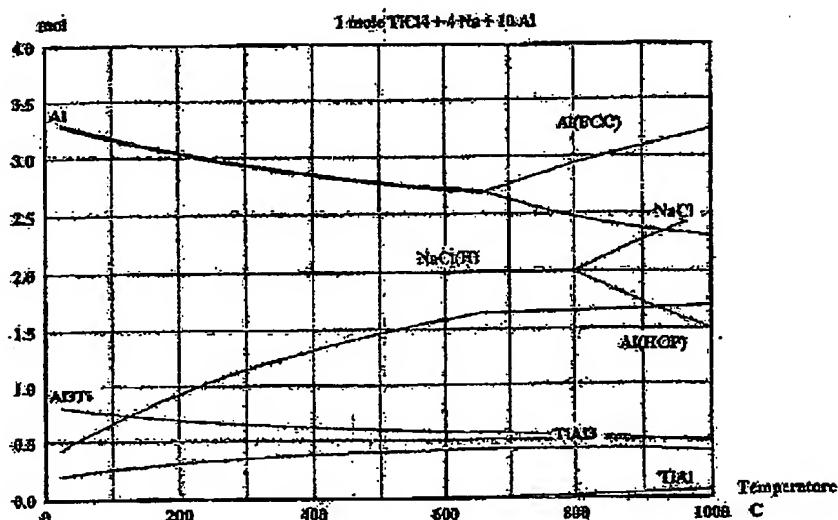
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Figure 1: Equilibrium composition for a mixture of 1 mole  $\text{TiCl}_4$  + 2 moles  $\text{Mg}$  + 2 moles  $\text{Al}$ .Figure 2: Equilibrium composition for a mixture of 1 mole  $\text{TiCl}_4$  + 4 moles  $\text{Na}$  + 2 moles  $\text{Al}$ .

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Figure 3: Equilibrium composition for a mixture of 1 mole  $TiCl_4$  + 4 moles  $Na$  + 10 moles  $Al$ .

I further declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: May 1<sup>st</sup>, 2009

  
Jawad Haider